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a. REPORT UU	b. ABSTRACT UU	c. THIS PAGE UU			Peter Burke
					19b. TELEPHONE NUMBER 949-824-9326

Report Title

Final Report: Nanoscale Imaging Technology for THz Frequency Transmission Microscopy

ABSTRACT

final progress report

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

<u>Received</u>	<u>Paper</u>
08/29/2012 15.00	Peter J. Burke, Douglas C. Wallace, Antonio Davila Jr, Katayoun Zand, Tae-Sun Lim. Wafer-scale mitochondrial membrane potential assays, Lab on a Chip, (04 2012): 2719. doi: 10.1039/c2lc40086c
08/29/2013 27.00	Peter J. Burke, Nima Rouhi, Yung Yu Wang. Ultrahigh conductivity of large area suspended few layer graphene films, Applied Physics Letters, (12 2012): 0. doi: 10.1063/1.4772797
08/29/2013 28.00	Peter J. Burke, Yung Yu Wang. A large-area and contamination-free graphene transistor for liquid-gated sensing applications, Applied Physics Letters, (07 2013): 0. doi: 10.1063/1.4816764
08/29/2013 29.00	Katayoun Zand, Ted Pham, Antonio Davila, Douglas C. Wallace, Peter J. Burke. Nanofluidic Platform for Single Mitochondria Analysis Using Fluorescence Microscopy, Analytical Chemistry, (06 2013): 0. doi: 10.1021/ac4010088
10/28/2014 33.00	Yung Yu Wang, Peter J. Burke. Polyelectrolyte multilayer electrostatic gating of graphene field-effect transistors, Nano Research, (09 2014): 0. doi: 10.1007/s12274-014-0525-9
10/28/2014 35.00	Weidong Zhang, Phi H. Q. Pham, Elliott R. Brown, Peter J. Burke. AC conductivity parameters of graphene derived from THz etalon transmittance, Nanoscale, (09 2014): 13895. doi: 10.1039/C4NR03222E
10/28/2014 34.00	Ted D. Pham, Yung Yu Wang, Katayoun Zand, Jinfeng Li, Peter J. Burke. Charging the Quantum Capacitance of Graphene with a Single Biological Ion Channel, ACS Nano, (05 2014): 4228. doi: 10.1021/nn501376z
10/30/2013 32.00	Nima Rouhi, Santiago Capdevila, Dheeraj Jain, Katayoun Zand, Yung Yu Wang, Elliott Brown, Lluís Jofre, Peter Burke. Terahertz graphene optics, Nano Research, (09 2012): 0. doi: 10.1007/s12274-012-0251-0

TOTAL: 8

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received

Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. Ted Pham “Charging the Quantum Capacitance of Graphene with a Single Biological Ion Channel” UC System Wide Bioengineering Symposium, June 2014, Irvine, CA (poster presentation).
2. Katayoun Zand “Nanochannel Trap Arrays For Monitoring Single Mitochondrion Behavior” UC System Wide Bioengineering Symposium, June 2014, Irvine, CA.
3. Peter Burke “Towards a Single Cell Radio”, ACM NANOCOM, May 13 – 14, 2014, Atlanta, Georgia.
4. Phi Pham “Broadband Measurement of Terahertz Transmission Modulation Using Back-Gated Graphene”, MRS Spring meeting 2014, April 2014, San Francisco, CA.
5. Weiwei Zhou “Integration of Carbon Nanotube Network Transistor and Tethered Lipid Bilayer on SiO₂ surface for Single-Ion Channel Recording”, MRS Spring meeting 2014, April 2014, San Francisco, CA. (poster presentation).
6. Will Wang “Charging the Quantum Capacitance of Graphene with a Single Biological Ion Channel”, MRS Spring meeting 2014, April 2014, San Francisco, CA.
7. Katayoun Zand “Nanochannel Devices for Single Mitochondrion Membrane Potential Assays” MRS Spring meeting 2014, April 2014, San Francisco, CA.
8. Will Wang “Charging the Quantum Capacitance of Graphene with a Single Biological Ion Channel”, Biophysical Society 58th Annual Meeting, February 2014, San Francisco, CA. (poster Presentation)
9. Katayoun Zand “Nanochannel Trap Arrays For Monitoring Single Mitochondrion Behavior”, Biophysical Society 58th Annual Meeting, February 2014, San Francisco, CA.
10. Peter Burke “Nanochannel Trap Arrays for Monitoring Single Mitochondrion Behavior”, NCI-NIBIB Point of Care Technologies for Cancer Conference, January 8-10, 2014, Bethesda, Maryland
11. Peter Burke “THz Wireless Communication”, IEEE Radio and Wireless Symposium, January 21, 2014, Newport Beach, CA
12. Peter Burke “Nanofluidic Platform for Single Mitochondria Analysis Using Fluorescence Microscopy”, 21th ECDO Euroconference on Apoptosis, September 25-28, 2013, Paris, France.

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received

Paper

08/29/2013 30.00 Katayoun Zand, Ted Pham, Antonio Davila Jr, Douglas C. Wallace, Peter Burke. NOVEL APPROACH TOWARDS TRAPPING AND IMAGING OF INDIVIDUAL MITOCHONDRIA, Conference on Microtechnologies in Medicine and Biology . 10-APR-13, . : ,

TOTAL: 1

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received

Paper

08/29/2012 16.00 Tae-Sun Lim, Dheeraj Jain , Peter J. Burke. Biomembrane Gated Carbon Nanotube Transistor as a Sensing Platform, Micro TAS 2011. 02-OCT-11, . : ,

08/29/2012 17.00 Nima Rouhi, Dheeraj Jain, Peter John Burke. Radio frequency nanoelectronics based on carbon nanotubes, SiRF 2012. 16-JAN-12, . : ,

08/30/2012 18.00 Nima Rouhi, Dheeraj Jain, Santiago Capdevila, Lluís Jofre, Elliott Brown, Peter J. Burke. Broadband Conductivity of Graphene from DC to THz, IEEE Nano 2011. 15-AUG-11, . : ,

08/30/2012 19.00 Tae-Sun Lim, Dheeraj Jain, Peter J. Burke. Fabrication of Supported Lipid Bilayer (SLB) and Nanotube Transistor Hybrid Biosensing Platform Using Microfluidic Channels, IEEE Nano 2011. 15-AUG-11, . : ,

08/30/2012 20.00 Dheeraj Jain, Nima Rouhi, Peter J. Burke. Novel Approach Towards Performance Enhancement of All Semiconducting Carbon Nanotube Devices for Printed Electronics, IEEE Nano 2011. 15-AUG-11, . : ,

TOTAL: 5

(d) Manuscripts

<u>Received</u>	<u>Paper</u>
08/29/2012 14.00	Nima Rouhi, Dheeraj Jain, Peter John Burke. High Performance Semiconducting Nanotube Inks: Progress and Prospects, ACS Nano (09 2011)
08/30/2012 22.00	Peter Burke, Nima Rouhi, Santiago Capdevila, Dheeraj Jain, Katayoun Zand, Yung Yu Wang, Elliott Brown, Lluís Jofre. Terahertz Graphene Optics, Nano Research (08 2012)
08/30/2012 23.00	Katayoun Zand, Ted Pham, Antonio Davila Jr, Douglas C. Wallace, Peter J. Burke. Nanofluidic Platform for Single Mitochondria Analysis, Nature Methods (08 2012)
09/14/2010 1.00	N. Rouhi, D. Jain, K. Zand, P. J. Burke. Fundamental Limits on the Mobility of Nanotube-Based Semiconducting Inks, (09 2010)
09/14/2010 2.00	D. Jain, N. Rouhi, C. Rutherglen, C. G. Densmore, S. K. Doorn, P. J. Burke. Effect of source, surfactant, and deposition process on electronic properties of nanotube arrays, (09 2010)
09/14/2010 3.00	N. Rouhi, D. Jain, P. J. Burke. Carbon Nanotube Devices for RF Applications, (09 2010)
09/14/2010 4.00	Peter Burke & Christopher Rutherglen. Towards a single-chip, implantable RFID system: is a single-cell radio possible?, (01 2010)
09/14/2010 5.00	Sungmu Kang, Chris Rutherglen, Nima Rouhi, Peter Burke, L. N. Pfeiffer, and K. W. West. An RF Circuit Model of a Quantum Point Contact, (03 2010)
09/14/2010 7.00	N. Rouhi, D. Jain, K. Zand, P. J. Burke. Semiconducting-Enriched Printed Carbon Nanotube Mat used for Fabrication of Thin Film Transistors, (09 2010)
09/14/2010 10.00	Tae-Sun Lim, Antonio Dávila, Douglas C. Wallace, and Peter Burke. On-Chip Ion-selective Microsensor for Evaluation of Mitochondrial Membrane Potential , (09 2010)
09/14/2010 11.00	N. Rouhi, D. Jain, K. Zand, P. J. Burke. All-Semiconducting Nanotube Devices for RF and Microwave Applications, (09 2010)
09/14/2010 12.00	Tae-Sun Lim, Antonio Dávila, Douglas C. Wallace and Peter Burke. Assessment of mitochondrial membrane potential using an on-chip microelectrode in a microfluidic device , (04 2010)

TOTAL: 12

Number of Manuscripts:

Books	
<u>Received</u>	<u>Book</u>
TOTAL:	

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

News: Tuning in to Graphene, Communications of the ACM, October, 2013

Innovator of the Year Award, HSSOE, UCI, 2014 (Peter Burke)

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>	Discipline
Phi Pham	0.18	
Yung-Yu Wang	0.15	
FTE Equivalent:	0.33	
Total Number:	2	

Names of Post Doctorates

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Names of Faculty Supported

NAME

PERCENT SUPPORTED

National Academy Member

Peter Burke

0.18

FTE Equivalent:

0.18

Total Number:

1

Names of Under Graduate students supported

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:..... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):..... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:..... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:..... 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME

PERCENT SUPPORTED

FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

see attached

Technology Transfer

see attached

Final Progress Report for
Grant # W911NF-09-1-0319
Prepared October 8, 2014

Submitted to Dr. Joe Qiu by:

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1. History and background of grant

This project is an outgrowth of a DTRA funded feasibility study for the development of a nanoscale THz spectrometer. The goal of this 3-year grant is to make progress towards fabrication and demonstration of a high resolution method to use nanotechnology (specifically nanotubes and graphene) to measure THz signals.

The grant was funded for 6 mos. (\$70,000 for 07/01/2009-12/31/2009), then another 6 mos. (\$68,592 for 01/01/2010-06/30/2010). Option #2 was exercised with incremental funding in the amount of \$60,000 in November, 2010 of the \$137,463 budgeted for 12 mos. during 07/01/2010-06/30/2011. In June 2011, work on option #2 totaled \$75,600, while the amount funded was \$60,000. It was anticipated that option #2 will be additionally funded for at least \$15,600 or more. An additional \$40,000 was exercised for Option #2 to the project in March 2012. In June 2012, a No-Cost Time extension was granted to keep the project active through June 30, 2013. The last Interim Progress Report covered the period up to July 31, 2012.

In February 2013, additional funding of \$37,463 were added to the grant. In March, 2013, the final option of \$142,465 was added and the period of performance extended to June 30, 2014.

The previous IPR described the fourth year progress on this grant (for the period beginning August 01, 2012 and ending July 31, 2013.)

Prior IPRs were submitted that covered the previous periods of performance ending 7/31/2013. This report contains them implicitly, as well as reports on additional progress for the period of performance 8/1/2013-6/30/2014.

2. Budgetary issues

The federal government shut down in the fall of 2013, and we had already committed to hiring an engineer to run our nanotube and graphene synthesis furnace, and two students. A related MURI effort was unfunded during part of the shut down, so we leveraged the ARO core

for this work. This provided a very synergistic combination of materials growth for THz antennas based on nanomaterials. (See task 5 in the next section).

3. Progress this year

As we described in our last IPR, we have executed the plans for the final year of this project. We continued to develop graphene materials for THz detection applications. The bulk of the effort focused on materials synthesis and characterization in order to supply devices for THz spectroscopy.

We also hired an engineer to run a commercial nanotube furnace that can make extremely dense aligned arrays of nanotubes, which was purchased by UCI at a deep discount.

The original grant was to use double quantum dots for THz camera, however the project progress has been significantly enhanced because of our realization the graphene can actually act to guide THz waves and modulate them. This was one of the original tasks, #5, called “antenna aperture issues”, in which we stated the problem but not the solution of how to guide THz waves from free space onto any kind of nanodevice. Our realization that graphene can be a waveguide and hence possibly guide free space waves onto nanodevice that are actually made of graphene themselves will be further explored in this project. However to make good THz waveguides with plasmonic effects one needs high quality material (high mobility).

Therefore, this was an important focus (to develop better mobility, large area graphene) for THz wave modulation for applications in THz cameras for biosensing in this last year.

Graphene synthesis and device processing (Irvine)

During the period, we developed dramatically improved graphene quality over large area. In the beginning of the period, we had a Dirac voltage of graphene on silicon of about 100 V. Now it is down around a few volts, which allows for higher transconductance. In addition, the on/off ratio was improved from about 1.1:1 to about 5:1. Finally, the lowest resistance was improved from a few k Ω per square to about 1 k Ω per square. To be sure, these metrics are not new to graphene. However, to our knowledge, this is the first time they have been achieved in large area graphene. (A well publicized study of large area graphene by Samsung [3] was not gated, as we need for the THz modulator). Our graphene is about 1 cm x 1 cm.

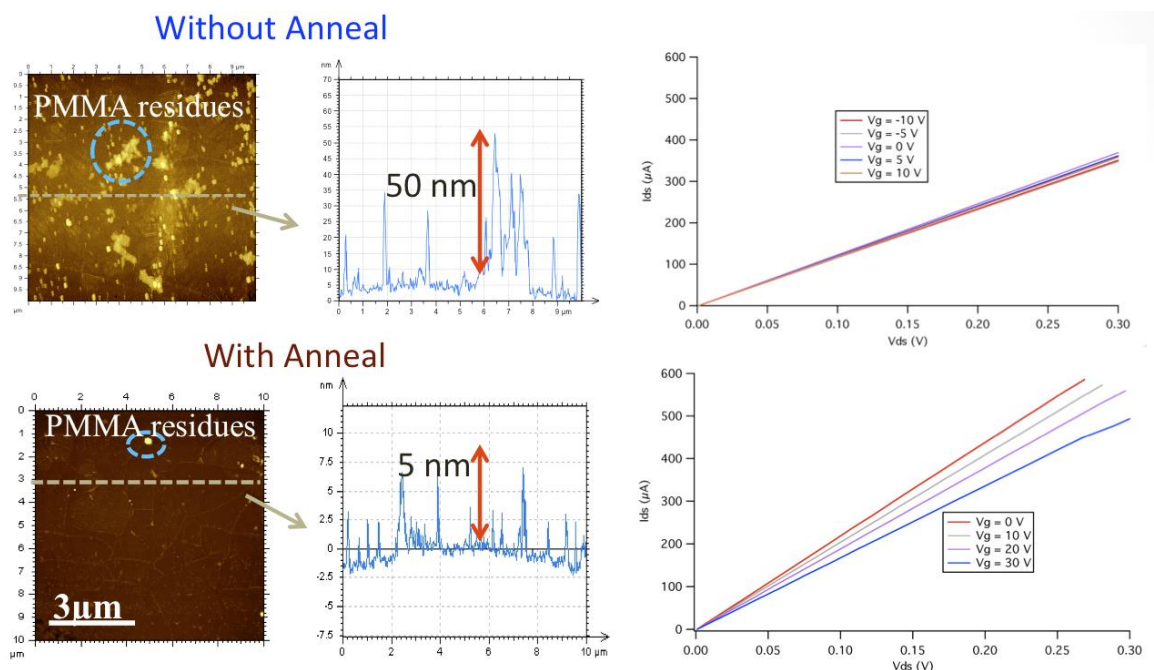


Figure 1: Effects of PMMA removal using vacuum annealing

Much of the actual work done in the first half of the period was to develop an annealing procedure to remove residual PMMA left over from the transfer process as seen in Figure 1. We performed 34 anneal runs during the period on our First Nano CVD system (Figure 2), and were able to develop an appropriate PMMA removal recipe, which consists of 200 sccm H₂ and 200 sccm Ar at 400 degrees Celsius at 500 mTorr for one hour.



Figure 2: EasyTube3000 CVD System used for annealing and graphene growth

During the second half of the period, we were able to change one of the mass flow controllers on the First Nano CVD system for one with a low-max flow rate, allowing for the successful synthesis of single layer graphene with quality comparable to other commercially available graphene suppliers as displayed in Figure 3. This work performed under this ARO core grant, which allowed for the hiring of a senior engineer for 3 months (Lei Ding, Oct-Dec 2013) in order to get the furnace running for graphene.

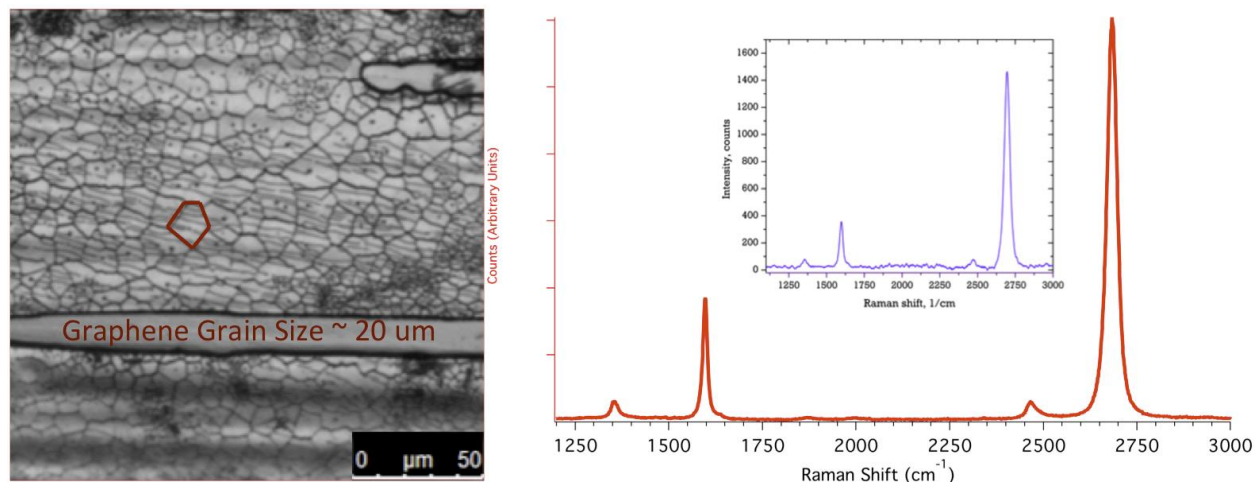


Figure 3: Optical microscopy image of transferred graphene film ($\sim 20 \mu\text{m}$ grain size). The Raman spectra of UCI grown graphene indicates monolayer (inset shows Raman spectra from Graphene Super Market for comparison)

The transferred graphene still had a large Dirac point ($\sim 100 \text{ V}$), regardless of whether it was graphene grown in our lab, or provided by a commercial supplier (Graphene Supermarket from New York, Graphenea from Europe, and Graphene Sq from Korea). In order to address the issue of a large Dirac voltage, we proceeded to modify the substrate surface using chemical functionalization of self-assembled monolayers of octadecyltrichlorosilane (OTS) as described in [4]. Using this technique, we were able to decrease the Dirac point from somewhere near 100 volts to a voltage near 10 volts as shown in Figure 4. Although low Dirac voltages for GFETs and substrate modification using OTS have both been previously demonstrated, to our knowledge, we are the first to demonstrate the use of these methods for centimeter scale device areas.

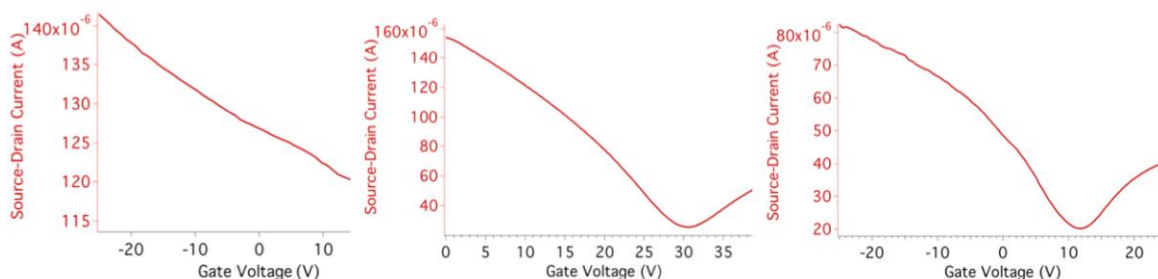


Figure 4: Evolution of Dirac point over time. The first chart shows devices with a unobservable Dirac point ($> 100 \text{ V}$). In the following charts, we can see devices with Dirac point lowered to 30 V, and 10 V.

4. Relationship of this project to MURI project

A separate MURI project is funding work on biosensors (W911NF-09-1-0319). The work reported in this period of performance was mostly materials development and graphene transfer methods, and so is aimed at the ARO CORE project but has additional benefits to the MURI effort. Note that while this report is similar to the MURI IPR, this work is nanomaterials synthesis development and the MURI report is more focused on the applications of the nanomaterials. Still because they are closely related the IPR and this report are used to show the synergies.

The work on nanomaterials development was a continuous process development that was not halted or started up based on spurts in funding. Instead, to keep the development on track, during the Fall 2013 the process development was supported by the ARO core, whereas during the spring 2014, this work was leveraged to continue improving materials and also to deliver the testbeds to the MURI participants. Therefore, some of the graphs in this report overlap with the once in the MURI IPR.